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IS 12030 (1986): Semi-Acid Oil [FAD 13: Oils and Oilseeds]



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Indian Standard
SPECIFICATION FOR
SEMI-ACID OIL

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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Indian Standard

SPECIFICATION FOR SEMI-ACID OIL

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Indian Standard

SPECIFICATION FOR SEMI-ACID OIL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 28 November 1986, after the draft finalized by the Oils and Oilseeds Sectional Committee had been approved by the Chemical Division Council and the Agricultural and Food Products Division Council.

0.2 An alternative to making acid oil is the semi-acid oil. If the quantity of mineral acid used to split the soap stock is kept to around 90 percent of the theoretical value, an organic layer can be separated which contains very little of mineral acids, but still contains some soap and therefore, it is not corrosive. The bulk of the water is removed and, therefore, transportation becomes cheaper than soap stocks. Semi-acid oil preserves much better than soap stock and, therefore, has some of the advantages of the acid oil, namely, high concentration of total fatty matter (TFM) and some advantages of soap stocks (non-corrosive nature). Semi-acid oil, if intended for distilled fatty acids, needs further processing to get a complete split.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the results of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for semi-acid oil.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS : 11476-1985† shall apply.

*Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to oils and fats.

3. REQUIREMENTS

3.1 The semi-acid oil shall have an iodine value as agreed to between the purchaser and the supplier.

3.2 The semi-acid oil shall also conform to the requirements given in Table 1, when tested according to methods prescribed in col 4, 5 and 6 of Table 1.

TABLE 1 REQUIREMENTS FOR SEMI-ACID OIL

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO CL No. OF		
			IS : 286- 1978*	IS : 548 (Part 1)- 1964†	Appendix A
(1)	(2)	(3)	(4)	(5)	(6)
i)	Total fatty matter (TFM), percent by mass, <i>Min</i>	85	15	—	—
ii)	Moisture and volatile matter content, percent by mass, <i>Max</i>	10	4	—	—
iii)	pH of 1 percent solution, <i>Min</i>	6.0	—	—	A-2
iv)	Unsaponifiable matter, per- cent by mass on TFM, <i>Max</i>	5.0	—	8	—
v)	Oxidized fatty acid, percent by mass on TFM, <i>Max</i>	5.0	—	—	A-3
vi)	Iron, ppm, <i>Max</i>	500	—	—	A-4
vii)	Ash, percent by mass, <i>Max</i>	0.25	—	—	A-5

*Methods of sampling and test for soaps (*second revision*).

†Methods of sampling and test for oils and fats: Part 1 Sampling, physical and chemical tests (*revised*).

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in suitable containers or transported through tankers or as agreed to between the purchaser and the supplier.

4.2 Marking — The material, if packed in containers, shall be securely closed and marked with the following information:

- Name of the material;
- Manufacturer's name and recognized trade-mark, if any;
- Net mass of the material;
- Batch number or lot number in code or otherwise; and
- Month and year of manufacture.

4.2.1 The containers shall also, in addition, be legibly and indelibly marked with the information required under the *Standards of Weights and Measures (Package Commodities) Rules, 1977*.

4.2.2 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed under 3 of IS : 548 (Part 1)-1964*.

APPENDIX A

(Table 1, Sl No. iii, v, vi, and vii)

METHODS OF TEST FOR SEMI-ACID OIL

A-1. QUALITY OF REAGENTS — Unless stated otherwise, pure chemicals and distilled water (see IS : 1070-1977†) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF pH

A-2.1 General

A-2.1.1 pH determination should be made in an acid-free atmosphere.

A-2.2 Apparatus

A-2.2.1 pH Meter — any standard electrometric instrument, equipped with a low sodium error glass electrode. The instrument shall be calibrated and standardized with standard buffer solutions (see A-2.3.2) before use (see IS : 2711-1979‡).

*Methods of sampling and test for oils fats: Part 1 Sampling, physical and chemical tests (revised).

†Specification for water for general laboratory use (second revision).

‡Specification for direct reading pH meter (second revision).

A-2.2.2 Volumetric Flask — 1 000-ml capacity.

A-2.2.3 Beaker — 100-ml capacity.

A-2.3 Reagents

A-2.3.1 Distilled Water — Distilled water shall be boiled thoroughly or purged with carbon dioxide-free air to remove carbon dioxide, and shall be protected with soda lime or soda asbestos while cooling and in storage. The pH of this water shall be between 6.2 and 7.2 at 27°C. The residue on evaporation when heated at 105°C for one hour shall not exceed 0.5 mg per litre.

A-2.3.2 Standard Buffer Solution — Any two suitable buffer solutions within the pH range of 9 to 11 at 27°C for calibrating the pH meter.

A-2.4 Procedure

A-2.4.1 Weigh 10 ± 0.001 g of the material and transfer to a 1-litre volumetric flask. Partially fill the flask with distilled water and agitate until the sample is completely dissolved. Adjust the temperature of the solution and the distilled water to $27 \pm 0.5^\circ\text{C}$, and fill to the calibration mark with the distilled water. Stopper the flask, mix thoroughly, and allow the solution to stand at a temperature of 27°C for 2 hours prior to measuring the pH. Measure the pH of the solution using a glass electrode.

A-3. DETERMINATION OF OXIDISED FATTY ACID

A-3.0 General

A-3.0.1 A known quantity of the material is saponified with alcoholic potash and the soap formed is treated with mineral acid to release the fatty acids. The petroleum ether insoluble but ethyl ether soluble matter, which is termed as oxidized fatty acid, is determined by evaporation of the ethyl extract of the residual aqueous layer left behind after the petroleum ether extraction.

A-3.1 Apparatus

A-3.1.1 Stoppered Glass Cylinder — 250-ml capacity.

A-3.1.2 Separating Funnel — 500-ml capacity.

A-3.1.3 Flasks — 250-ml capacity, flat bottom; and 300-ml capacity, conical.

A-3.2 Reagents

A-3.2.1 Alcoholic Potassium Hydroxide Solution — Dissolve 50 g of potassium hydroxide in 1 litre of 95 percent ethyl alcohol.

A-3.2.2 Dilute Hydrochloric Acid — 1 : 1 by volume.

A-3.2.3 Methyl Orange Indicator — Dissolve 0.1 g of methyl orange indicator in 100 ml of water.

A-3.2.4 Petroleum Ether — 60°C/80°C distillation range.

A-3.2.5 Ethyl Ether

A-3.3 Procedure

A-3.3.1 Weigh accurately 3 to 5 g of the fatty matter into a 300-ml conical flask. Add 50 ml of alcoholic potash, cover with an inverted funnel and heat on a water-bath to saponify. Agitate frequently and heat for at least 30 minutes or until saponification is complete.

A-3.3.2 Remove the watch-glass and continue heating on a water-bath with occasional agitation to evaporate the alcohol. To avoid oxidation, do not evaporate beyond a pasty mass. If necessary, add a small amount of water when most of the alcohol has evaporated.

A-3.3.3 Add 100 ml of distilled water and heat until the soap has completely dissolved. Wash the contents into a glass-stoppered cylinder with hot distilled water, taking care not to exceed a total volume of 130 ml in the cylinder.

A-3.3.4 Add 3 to 5 drops of indicator and neutralize with hydrochloric acid to the pink methyl orange end point. Then add 1 ml of excess acid. Rotate the cylinder gently to mix the contents.

A-3.3.5 Cool to at least 35°C and add 125 ml of petroleum ether. The fatty acids need not have cleared completely before adding the ether. Stopper the cylinder, shake gently and allow to stand until the petroleum ether layer separates.

A-3.3.6 Siphon the petroleum ether layer into a 500-ml separating funnel, making sure that as little as possible of the insoluble matter which gathers at the ether-water interface is carried over into the separating funnel. If any appreciable amount of insoluble matter does siphon over into the separating funnel, it will usually settle to the bottom and shall be drained back into the extraction cylinder. Make at least 4 more similar extractions using 25 to 30 ml of petroleum ether, shaking the cylinder vigorously for 30 seconds with each extraction. Extractions shall be continued until the petroleum ether layer is practically colourless.

A-3.3.7 To the acid water remained in the extraction cylinder add 25 to 30 ml of ethyl ether, stopper, shake gently and allow to stand until the ether layer separates. Siphon the ethyl ether layer through a filter

paper into a tared 250-ml flat bottom flask which has been dried and cooled in a desiccator. Make at least 4 more similar extractions using 25 to 30 ml of ethyl ether each time, and shaking the cylinder vigorously for 30 seconds with each extraction. The last ethyl ether extract shall be practically colourless.

A-3.3.8 Filter all extracts through the same filter paper and finally wash this filter paper thoroughly with ethyl ether to recover all the oxidized acids.

A-3.3.9 Evaporate the ethyl ether extracts on a water-bath under a gentle stream of clean dry air. Finally, dry the oxidized fatty acids in air oven at $105 \pm 2^\circ\text{C}$ for 30 minutes. Cool in a desiccator to room temperature and weigh. Repeat until constant mass, that is, to within 0.1 percent between successive weighings, is obtained.

A-3.4 Reporting — Report the oxidized fatty acids as a percentage of the material taken for the test.

A-4. DETERMINATION OF IRON CONTENT

A-4.0 Outline of the Method

A-4.0.1 The method is based on the isolation of metal by dissolving in hot water. The aqueous extract is treated with citric acid to sequester aluminium and then thioglycolic acid in ammoniacal solution is added and colour measured spectrophotometrically.

A-4.1 Apparatus

A-4.1.1 Spectrophotometer

A-4.2 Reagents

A-4.2.1 Liquor Ammonia — Relative density 0.9.

A-4.2.2 Dilute Sulphuric Acid — 50 percent (v/v).

A-4.2.3 Citric Acid (Aqueous Solution) — 50 percent (v/v).

A-4.2.4 Thioglycolic Acid (Aqueous Solution)

A-4.2.5 Standard Iron Solution — Containing $10\ \mu\text{g}$ of iron per ml prepared from ferric ammonium sulphate $[\text{Fe}_2(\text{SO}_4)_3 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 12\text{H}_2\text{O}]$ in acid solution.

A-4.2.6 Methyl Red Indicator — 0.1 percent aqueous solution.

A-4.3 Procedure

A-4.3.1 Isolation of Metals — Weigh 50 g of the sample in a beaker and dissolve it with hot water. To this solution add 40 ml of concentrated hydrochloric acid with constant stirring and keep the beaker on